

# Monday Afternoon, October 18, 2010

## Vacuum Technology

Room: Laguna - Session VT+MS-MoA

### Gas Analysis in Vacuum and Process Applications

Moderator: S. Thornberg, Sandia National Laboratories

2:00pm **VT+MS-MoA1 Miniature Mass Spectrometers and Ambient Analysis by Mass Spectrometry**, *R.G. Cooks, R.J. Noll, Z. Ouyang*, Purdue University **INVITED**

Two inter-related areas of rapid growth in mass spectrometry are i) Ambient ionization and ii) miniature mass spectrometers. This talk covers both topics. It attempts to provide the conceptual basis for these developments as well as summarizing the state of the technology and citing typical applications.

The authors believe that mass spectrometers are in the early stages of a radical transformation that will make them much more versatile and much more widely applied in situ than in the laboratory in future. At the same time they will increasingly be operated by non-technical staff including nurses, production line workers, industrial hygiene and food safety inspectors, surgeons and others who are not skilled in scientific instrumentation. The essential features needed for these developments are already in place, namely the rapid growth of regulatory and other demands for chemical analysis and the laborious nature of current standard laboratory methodology in many areas of chemical analysis.

Miniature and micro mass spectrometers are of great current interest. For many reasons, quadrupole ion traps are the most appropriately miniaturized mass analyzers and both microscale (10's of microns) and miniscale (100's of microns) instruments have been used. Arrays of such analyzers have further advantages in terms of reduction in power requirements. Most attention has gone to the mass analyzer but full systems (sampling, ion source, data capture and reduction) have been built and will be discussed, including a series of Mini mass spectrometers built in our labs. These handheld mass spectrometers have good performance (unit mass resolution to  $m/z$  500) and allow gases, solutions and surfaces to be analyzed using a variety of ionization methods including internal electron impact and external electrospray ionization and desorption electrospray ionization (DESI). These systems are pressure-tolerant, they provide tandem mass spectrometry capabilities and satisfies critical size and weight criteria while providing essentially instantaneous chemical analysis.

Ambient ionization - in which samples are examined without preparation in their native state is readily applied with miniature mass spectrometers. These methods, of which DESI is the prototypical example, do not require sample preparation and operate in the ambient environment. High throughput analysis of complex mixtures - with tandem mass spectrometry being used to resolve the overlapping chemical signatures - is possible. Examples range from benzene vapor in urban air to agrochemical residues in produce to phospholipid distributions in diseased tissue.

2:40pm **VT+MS-MoA3 Accurate Determination of Molar Quantity for Gas in a Vacuum Chamber with Extreme Temperature Variations**, *H.C. Peebles, M.S. Benner*, Sandia National Laboratories, *T.K. Mehrhoff*, Independent Contractor to Sandia National Laboratories

Mass spectroscopy is routinely used to quantify gases in analytical measurements and process diagnostics. In many of these applications, gases enter the ion source of the mass spectrometer through a molecular leak. Sections of the gas manifold on the high pressure side of the leak may contain extreme temperature gradients such as a gas source operating at a very high temperature or a cryogenic trap at low temperature with the remaining manifold components near room temperature. Calibration of the mass spectrometer response to the molar quantity of gas present in the manifold must take into account the thermal gradient over pressures that can span the range from viscous flow to the molecular flow regime where thermal transpiration dominates. This paper will present a method for calibrating the molar quantity of gas present in a manifold with a large but constant temperature gradient using a pressure gauge and molar calibration source (MCS) attached to a room temperature section of the manifold. The MCS is a calibrated gas volume and integrated pressure gauge maintained at constant temperature. Molar quantities of gas released from the MCS into the manifold are related to the response of the pressure gauge using empirical  $n/P$  functions. These functions accurately relate the manifold pressure to the molar quantity of gas in the manifold as long as the temperature distribution across the manifold remains stable. An example will be presented showing the application and stability of  $n/P$  functions used in thermal desorption measurements of hydrogen isotope concentrations in

thin metal films. The construction and critical performance characteristics of the MCS will also be described.

Sandia National Laboratories is a multi-program laboratory operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin company, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

3:00pm **VT+MS-MoA4 Hydrocarbon Measurements at ppb Level at 10Pa Absolute Pressure**, *R. Versluis, M.F. Dekker*, TNO Science and Industry, Netherlands

In Extreme UltraViolet lithography (EUVL), 13.5 nm light is used for imaging the reticle pattern onto the wafer. Since EUV light is absorbed by all materials (including gases) a reflective optical system is used in EUV wafer steppers. Wafer resist outgassing during exposure is one of the most important contributors to mirror contamination. Just one or a few monolayers of Carbon on the multi-layer reflective optics of the system leads to an unacceptable reflectivity loss. Considering the fact that a large fraction of the EUV induced wafer resist outgassing constitutes of hydrocarbons, suppression and mitigation of these molecules is absolutely necessary.

Different methods exist to suppress hydrocarbons and other contamination from wafer, or 'dirty' components. In order to qualify such methods, very sensitive hydrocarbon measurements need to be done at relatively high pressures.

We built a test rig capable to detect hydrocarbons at ppb level. The hydrocarbon levels that need to be measured are at the order of  $1E-9$  Pa, while the absolute pressure is at the order of 10 Pa. The test rig therefore needs to be extremely clean during the measurements. The test rig includes six large Turbo Molecular Pumps to keep the test rig at an absolute pressure of 10 Pa, while the component under test is continuously being flushed. The electro-polished setup (about 4 meters long and one meter high) can be completely baked at 150°C to reach the extreme clean conditions needed to measure the low concentrations of hydrocarbons. Measurements are done with a very sensitive residual gas analyser (RGA) which can be baked to 200°C. The RGA has been optimized in such a way that the sensitivity for heavy gases is increased with respect to the sensitivity for lighter gases (patent appointed). Test gases (hydrocarbons, Ar, N<sub>2</sub>) can be injected at different locations in the test rig. This way a contamination source in the wafer stage as well as wafer resist outgassing can be simulated.

With the test rig we can also do a thermal qualification of the component under test, to determine heat loads to the test component and heat load distributions.

The presentation will focus on design and engineering aspects of the test rig, the qualification of the test rig and the qualification of the component under test.

3:40pm **VT+MS-MoA6 Ratiometric and Absolute Partial Pressure Measurements with Low Mass Range Mass Spectrometers**, *G. Brucker, J. Rathbone, K. Van Antwerp, M.N. Schott*, Brooks Automation, Inc.

Low mass range mass spectrometers are routinely used to obtain partial pressure information in high vacuum and ultrahigh vacuum systems. Absolute and ratiometric partial pressure measurements are both applied to monitor and control vacuum processes and experiments. Mass spectrometers provide indirect partial pressure measurements, and require advanced data interpretation and analysis procedures in order to generate accurate partial pressure measurements from their raw spectral output. This presentation describes some of the modern methodologies used by commercial equipment manufacturers to derive accurate absolute and ratiometric partial pressure information using both quadrupole mass spectrometers and a new generation of electrostatic ion trap mass spectrometers. The advantages of combining accurate total pressure readings with native ratiometric partial pressure information from electrostatic ion traps is also described and compared to standard partial pressure measurement methodologies. Fast process control, in the millisecond timescale, based on partial and total pressure measurements is also described.

4:00pm **VT+MS-MoA7 Accurately Modeling the Natural Frequencies of Ions Ejected from an Anharmonic Resonant Ion Trap**, *M.N. Schott*, Brooks Automation, Inc.

A mathematical model is demonstrated that accurately predicts the natural frequencies of ions ejected from an Anharmonic Resonant Ion Trap (ion trap). The model is based upon a force balance equation of motion, which is comprised of a forcing function, a mass, a damper and a spring. The forcing function is a low amplitude RF signal, swept from high frequencies to low

frequencies over a given period, which locks-up, bunches and then ejects ions with a common mass-charge ratio using the principal of autoresonance. The ionized gas particles provide the mass portion of the equation, where specific autoresonant mass selection is dependent upon the selected ion's mass-charge ratio. The equation damping, or ion trap losses, are primarily dependent upon ion trap pressure. The spring in this model is provided by the trap's static anharmonic voltage gradient, which is a function of ion trap geometry and the associated relative-voltage-potentials distributed across the ion trap's geometric structures. The ejected ions natural frequencies are then proportional to the square root of the anharmonic voltage gradient, the pressure dependent damping and the autoresonant selected mass-charge ratio. Experimental results are presented that vary the independent anharmonic voltage gradient, pressure, and ionized gases versus dependent ejected ion natural frequencies on the ion trap compared with the model's predicted natural frequencies.

4:20pm **VT+MS-MoA8 Solving the Low Mass Range Mass Spectrometer Limitations (Zero Blast) using Electrostatic Ion Traps**, *P.C. Arnold, G. Brucker, J. Rathbone*, Brooks Automation, Inc.

The phenomena of typically unavailable resolution of low mass spectroscopic peaks (zero blast) will be presented along with a solution that allows clear resolution of those low mass peaks, for example, masses 1 to 4. A new mass spectrometer design composed of an electrostatic ion trap using anharmonic resonant trapping potentials will be shown to resolve this issue. The primary causes of poor low mass resolution will be presented. The background of the physical electronics of the problem will be discussed. The new mass spectrometer design will be described with respect to its effect on zero blast. A test program to demonstrate the solution and show results at scans of low mass will be presented.

4:40pm **VT+MS-MoA9 Ion Residence Times for Electron-Impact Ion Sources of Mass Spectrometers**, *R.E. Ellefson*, REVac Consulting, *M.F. Vollo*, INFICON, Inc.

Electron impact ionization is a common method of ion production for mass spectrometers. The mass spectrum produced is affected by the electron energy and ion residence time in the ionization region. The residence time is the time between the initial direct ionization event and the extraction and focus of the ion(s) from the ionization region into the mass analyzer. During the residence time, additional ionization, fragmentation and ion-molecule reactions can occur. In closed ion sources or other high-pressure ion sources, ion-molecule reactions can be significant; the ions extracted and analyzed can exhibit a bias in inferred gas composition due to the addition or depletion of the species-related ions of interest by the ion-molecule reactions. The presence of a potential well created by the ionizing electron beam and the ion extraction potentials dictate the residence time for ions in the ion source. Models for the depth of the potential well as a function of electron emission current and ion extraction potentials are given and estimated ion residence times are calculated. Evidence for change in residence time with emission current is provided by monitoring mass 80- $\text{Ar}_2^+$  abundance relative to mass 40- $\text{Ar}^+$  at a fixed ion source pressure for different emission currents. Other ion-molecule reactions (e.g.  $\text{N}_2^{+*} + \text{N}_2$  gives  $\text{N}_3^+ + \text{N}$ ) and charge-exchange reactions (e.g.  $\text{He}^+ + \text{Ar}$  yields  $\text{He} + \text{Ar}^+$ ) are presented together with pressure dependence and magnitude of the reactions. Recommendations are given for ion source operation to minimize biases in compositional analysis of gas mixtures.

5:00pm **VT+MS-MoA10 Performance Characteristics of a New Wide Range, Fast Settling Electrometer Design for a Residual Gas Analysis Mass Spectrometer**, *S. Billington*, MKS Spectra Products UK, *J. Blessing*, MKS Instruments, *R. Fletcher, P. Shaw*, MKS Spectra Products UK

The use of faraday or electron multiplier detectors in mass spectrometry has always presented the electronics engineer with the challenge of having to choose some compromises in a design for the electrometer. The maximum measurable signal usually determines the value for the feedback resistor that is required. However, with a conventional electrometer where a high impedance input Operational Amplifier is used in an inverting configuration with a feedback resistor, the noise is dominated by the feed-back resistor value. A typical quadrupole mass spectrometer design is capable of generating partial pressure ion currents from a scan of masses which encompasses the full range of the detector output. In order to utilise this full range, more than one feedback resistor is used with switching between gain ranges or a logarithmic amplifier is used. Intrinsically, the logarithmic amplifier requires significantly longer settling times for lower signal levels which tends to make it impractical for a mass spectrometer where two signals at the extremes of the dynamic range of measurement can be only a few milliseconds apart. Traditionally a gain switching electrometer has been the choice of RGA designers but this has resulted in the compromise of having to either choose which gain range to use for a particular scan of masses or wait for several tens of milliseconds each time the range is switched during the scan.

This work will describe a new electrometer design which allows the use of two gain ranges in a scan of masses with settling times of less than 20ms per measurement point. Data will be presented to show the effectiveness of the design for speed of measurement and the wide dynamic range available. RGA data will also be shown of common applications that traditionally would have required a compromised speed of acquisition or reduced dynamic range. Data will also be shown on the improved accuracy offered for fast transient peak measurements with the faster data acquisition rates of the new RGA design.

# Tuesday Morning, October 19, 2010

## Vacuum Technology

Room: Laguna - Session VT+MS-TuM

### Outgassing, Contamination Control, and Process Modeling

Moderator: M. Wuest, INFICON, Liechtenstein

8:00am VT+MS-TuM1 **Reduction of Hydrogen Content in Stainless Steel Vacuum Components**, *L.L. Wang, R.Y. Weinberg, K.A. Lao*, Los Alamos National Laboratory

Hydrogen is dissolved in stainless steel during the initial phases of production and fabrication. At room temperature, the dissolved hydrogen slowly diffuses out of the stainless steel. For stainless steel vessels assembled from commercially available vacuum components, we consistently measured constant rates of gas pressure increase in these sealed stainless steel vessels after they had been evacuated to  $1 \times 10^{-7}$  torr. The pressure in a 97 cc stainless steel vessel can reach up to 0.8 torr in six months at room temperature. The gas accumulated in these vessels, previously vacuum baked at 150°C for 48 hours to remove adsorbed gas, was analyzed to be essentially hydrogen. To determine how effective high-temperature vacuum bake out is in reducing the hydrogen content in the stainless steel components, we undertook a study that involved vacuum bakeout of the components at 400°C for 10 days and analysis of the hydrogen contents of the components with and without the vacuum bakeout. The hydrogen concentrations were measured by a LECO analyzer. The results will be presented and compared with that predicted by the Fick's law of diffusion.

8:20am VT+MS-TuM2 **Hydrogen Outgassing in a Small Vacuum Chamber**, *R.F. Berg*, National Institute of Standards and Technology

In a closed vacuum chamber, the problem of hydrogen outgassing from stainless steel increases with both the temperature and the chamber's surface-to-volume ratio. This talk will describe the outgassing in a chamber that is used to measure the vapor pressures of organic compounds in the range from 1 Pa to 100 kPa. The chamber, which is a small manifold built from stainless steel fittings and two capacitance diaphragm gauges, has a combination of challenges not usually present in a larger apparatus at room temperature. (1) Its volume of only 29 cm<sup>3</sup> created a relatively large surface-to-volume ratio. (2) Operating at temperatures as high as 200 °C greatly increased the outgassing rate. (3) The pressure gauges limited the maximum allowed bakeout temperature.

Closing the valve to the vacuum pump caused the pressure to increase nonlinearly with time. The initial rate slowed during several hours and usually became linear with time within one day. Intermittent pumping during one month at 200 °C showed that the linear rate decreased with an exponential time constant of approximately 11 days, which was consistent with the diffusion of hydrogen from the stainless steel fittings. Understanding this behavior is important because a pressure increase of 1 Pa/day ( $3 \times 10^{-10}$  Pa m<sup>3</sup>/s) can cause a significant error in the vapor pressure measurement. A model that accounts for the diffusion of hydrogen in the chamber wall and its nonlinear accumulation in the chamber volume will be compared to the pressure measurements.

8:40am VT+MS-TuM3 **Point-of-Use Abatement Devices and Exhaust Management Strategies**, *M. Sherer*, Sherer Consulting Services, Inc. **INVITED**

Semiconductor processes emit various contaminants which require exhaust management and in some cases point-of-use (POU) abatement. It is important to understand process exhaust management strategies, and to select the best, lowest cost-of-ownership POU abatement devices. This presentation will discuss these topics and provide relevant technical information.

9:20am VT+MS-TuM5 **Novel Instrument Capable of Efficient Gas Exchange to Remove Gas-phase Contamination in Complex Volumes Without Purging or High Vacuum**, *J. Brown, J. Hochrein, S. Thornberg*, Sandia National Laboratories

Countless systems used in research and in industry contain complex assemblies that are sealed in some type of enclosure, meant to isolate them from the harsh operating environment of the open atmosphere and to maintain a pristine internal atmosphere. Unfortunately, the internal atmosphere of any sealed component or system is, in the long-term, only as clean as the materials sealed within its enclosure. Over time, moisture or other volatile contaminants initially trapped in the materials can begin to

evolve and accumulate with potentially detrimental effects on the functionality of the component. This problem can be extremely difficult to address, depending on the physical and mechanical constraints of the particular system. Recently, an instrument was developed at Sandia National Laboratories that can "clean" the internal atmosphere of a critical optical component that cannot be subjected to conventional conditioning methods (such as N<sub>2</sub>/Ar purge, high-vacuum pumpdown, etc.). By using multiple pressurization and evacuation cycles tightly controlled within a narrow  $\pm 2$  psig window, the instrument fully and efficiently exchanges the liters of moisture- and contamination-laden internal gas of the component with clean, dry N<sub>2</sub>. This process is repeated as moisture from the internal materials diffuses back into the gas phase until, over time, the source of the moisture is depleted. This instrument has been successful in reducing the equilibrium gas-phase moisture levels in the optical component from the thousands of PPMv (parts per million by volume) to single-digit PPMv. This instrument, called the "Automated Pressure Cycler," will be discussed in detail.

10:40am VT+MS-TuM9 **Modeling, Design, Fabrication, and Characterization of a Pulsed Vacuum System**, *Z.C. Leseman, J. Butner*, University of New Mexico

Systems utilizing low to medium vacuum levels are becoming increasingly popular due to packaging of micro and nanoelectronic devices, exploration of surface phenomena, and gas-phase etching of materials. In this work, pulsed vacuum systems are modeled, designed, fabricated, and characterized. Modeling efforts focus on methods for calibration of volumes, pump-down / pressure-up times, and vacuum system configuration considerations. As a result of this systems of linear equations are developed and solved, as well as systems of coupled differential equations which are solved analytically and numerically (when necessary). As a result of this modeling effort a new method has emerged for vacuum processing at discrete pressures and discrete times. Experimental validation is presented in regards to specific applications: MEMS environmentally dependent stiction failure, vapor phase lubrication of MEMS, and XeF<sub>2</sub> vapor phase etching of Si.

11:00am VT+MS-TuM10 **Effects of Inlet Pipe Diameters on Pumping Performance of Turbomolecular Pump**, *F.-C. Hsieh, D.R. Liu, F.-Z. Chen*, National Applied Research Laboratories, Taiwan

The effects of inlet pipe diameters on pumping performance of turbomolecular pump (TP) are evaluated by commercial software VacTran. The result indicates that at inlet pipe diameter (D<sub>i</sub>) of 0.25 m the delivered pumping speed (DPS) of TP decreases from peak value (PV) of 680 L/s to about 430 L/s. However, DPS approaches to PV as D<sub>i</sub> increases to the largest one (1.0 m). Besides, the conductance of TP increases when D<sub>i</sub> increases. The conductance is proportional to the pipe radius at molecular flow. The pumping speeds (PS) versus the inlet pressure of TP and foreline (scroll pump) are evaluated. The PS for TP is higher than those obtained with foreline. Also, the DPS of TP is tested on an evaluation system constructed according to ISO-5302 standard. Good agreement between analysis and experimental data are shown. Finally, the throughput versus the inlet pressure of TP reveals linear trend in log-log scale within pressure range from  $3.1 \times 10^{-5}$  to  $9.3 \times 10^{-1}$  torr.

11:20am VT+MS-TuM11 **Optimal Configuration of a Radiometer Array for Low Pressure Applications**, *B. Cornella, A. Ketsdever*, University of Colorado at Colorado Springs, *N. Gimelshein, S. Gimelshein*, University of Southern California

A thin vane with a temperature gradient immersed in a rarefied gas will experience a force which tends to move the vane from the hot to the cold side. The radiometric force, as it is called, is the force that drives the Crookes radiometer. Applications of radiometric forces have been limited to date to high-density microdevices, most notably the atomic force microscope. However, applications can also involve larger devices in the low pressure regime (same equivalent Knudsen number). For an example, radiometric forces can act as a propulsion system to compensate disturbing forces on a vehicle traveling high in the atmosphere. Recent studies have shed new light on the relative influence of bulk radiometer area versus edge on force production, indicating that these effects are on the same order of magnitude in the Knudsen regime where the force is maximized (Kn=0.05). An experimental study has been conducted to investigate the impact of vane separation distance for a multiple-vane radiometer. This study is a first step in maximizing the force per unit volume (or mass) by optimizing the area versus edge geometries. Furthermore, this study provides experimental validation for today's numerical models involving rarefied radiometric flows. To emulate a near space environment, a 39" diameter vacuum chamber was used to set a range of pressures for the experiment from 0.1 to

10 Pa (corresponding to Knudsen numbers of 1.3 to 0.01). The experiment measures the total force of a one by three array radiometer configuration and compares it to a single vane with the same active area. Each individual radiometer vane consisted of a 40 mm square Peltier thermoelectric cooler where the temperature difference across the two surfaces was actively maintained at approximately 25 K. The relative separation between the vanes was varied from 0% (single vane setup) to 100% of the size of the individual vane element and the maximum forces between these varying configurations compared. Preliminary experimental results suggest that the total force produced by the overall radiometer increases with gap distance. Numerical results suggest that the optimum separation distance for maximum force production is around 75% of the vane height.

# Wednesday Morning, October 20, 2010

## Graphene Focus Topic

Room: Brazos - Session GR+EM+MS+TF+MI-WeM

## Graphene and Carbon-based Devices

Moderator: N.P. Guisinger, Argonne National Laboratory

8:00am **GR+EM+MS+TF+MI-WeM1 Epitaxial Graphene: Effects of Dielectric Overlayers and Device Design on FET Performance**, *M.J. Hollander, R. Cavalero, D. Snyder, M. LaBella, K. Trumbull, Z. Hughes, J. Robinson*, The Pennsylvania State University

The realization of a graphene-based electronic technology necessitates large-area graphene production, as well as the ability to integrate graphene with highly insulating films that act as the gate dielectric in field effect transistors (FETs). Graphene's two dimensional nature allows for phenomenal electronic properties and ultimate scalability, but also makes it susceptible to doping and scattering by charged impurities, dangling bonds, and other defects that may derive directly from choice in gate dielectric and deposition technique. The nature and extent of the effect of the dielectric over-layer on conduction within the graphene channel is of fundamental interest in designing and producing graphene based FETs. Atomic layer deposition (ALD) has proven to be an excellent technique toward the integration of dielectrics with graphene and provides a means to produce high quality films for gate dielectrics at temperatures below 300C, but requires the use of a thin nucleation layer to promote complete coverage and to protect the graphene.

We present results on graphene FETs utilizing various gate dielectrics and various nucleation layers. Graphene was grown epitaxially on 100 mm SiC wafers and processed using standard photolithographic techniques. Al<sub>2</sub>O<sub>3</sub> and HfO<sub>2</sub> gate dielectrics were investigated using SiO<sub>2</sub>, TiO<sub>2</sub>, and Al<sub>2</sub>O<sub>3</sub> nucleation layers in various combinations. We show that choice of gate dielectric and nucleation layer can have a dramatic effect on transistor performance and charge carrier mobility. Saturation current, transconductance, and device hysteresis were examined in the fabricated FETs while charge carrier mobility and charge carrier density within the epitaxial graphene were evaluated using Van der Pauw structures. Graphene FETs utilizing Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> seeded dielectrics exhibit the best performance while TiO<sub>2</sub> seeded and unseeded devices exhibit large gate leakage currents resulting in non-functioning FETs. Additionally we provide evidence that the choice of dielectric and seed can significantly impact the Dirac point (minimum conduction), amount of hysteresis, and on/off ratio of the graphene FETs. Trends in saturation current, and transconductance appear to be independent of nucleation layer and gate dielectric choice, indicating that conduction through the channel may be limited by mechanisms independent of the nucleation layer and gate dielectric.

In addition to the aforementioned performance metrics, FET performance after continued application of high electric fields across the channel will be reported. Finally, we examine how choice of channel length and width, along with transistor design, effect performance.

8:20am **GR+EM+MS+TF+MI-WeM2 Graphene Oxide on Patterned Self-Assembly Monolayer for Cancer Marker Detection**, *D.-J. Kim, C.Y. Jung, O.J. Yoon, N.-E. Lee*, Sungkyunkwan University, Republic of Korea, *J.-S. Park*, Korea Electronics Technology Institute, Republic of Korea

Graphene, two-dimensional sheet of sp<sup>2</sup>-hybridized carbon atom, has received much attention in recent years for many applications such as energy storage and conversion, batteries, fuel cells, optoelectronic device, field-effect transistors, electromechanical resonators, chemical sensors, and biotechnologies. Oxidized form of graphene, graphene oxide, which has functional groups of epoxide, carbonyls, hydroxide, and phenol groups at both sides can also provide various functionalities for biomedical applications because the high densities of defective sites can provide many active sites for electron transfer to biological species, similarly to graphene. In this study, we demonstrate the graphene oxide based immunosensor for cancer marker detection. Graphene oxide pattern was formed selectively on self-assembly monolayer (SAM) pattern formed by using inkjet printing. The hydroxyl terminated ITO electrode was first selectively functionalized by the amine (-NH<sub>2</sub>) group by inkjet printing of SAM, and the graphene oxide flakes were adsorbed selectively on the SAM pattern. After immobilization of monoclonal antibody to PSA (prostate specific antigen) which is the biomarker in prostate cancer, the electrochemical performance of immunosensor was evaluated by varying the concentration of PSA-ACT (prostate specific antigen/anti-chymotrypsin) complex. And the PSA polyclonal antibody conjugated with gold nanoparticles was applied to amplification of electrochemical signal because the attachment of

nanoparticles onto electrodes drastically enhances the conductivity and electron transfer from the redox analytes. The results showed that graphene oxide based immunosensor can be possibly applied to the delicate diagnosis of various disease markers as well as PSA at the extremely low level and with ultra-high sensitivity.

8:40am **GR+EM+MS+TF+MI-WeM3 Spintronics in Graphene Based Nanostructures**, *J. Fernández-Rossier*, Universidad de Alicante, Spain  
**INVITED**

Spintronics in graphene has received attention from two complementary points of view. On one side, the small spin orbit coupling and nuclear spin density suggest that graphene should have very long spin relaxation and decoherence times, which would make it suitable for long distance spin transport and, eventually, for spin-based quantum computing. Although small, spin orbit interaction turns graphene into a spin hall insulator, at very small temperatures, so that the edges of graphene should carry spontaneous spin currents. Independently of the above, the zigzag edges of graphene nanostructures, like graphene ribbons and islands, are predicted to present ferromagnetic spin order due to electron electron coupling. In this talk I will discuss the properties of graphene nanostructures when both the spin-orbit coupling and the Coulomb interactions are included. I will show how the combined action of spontaneous spin order and spin orbit coupling results in persistent charge currents flow in the edges of a variety of graphene nanostructures.

9:20am **GR+EM+MS+TF+MI-WeM5 CVD Gate Dielectrics and Bandgap Engineering of Graphene Layers**, *W.J. Zhu, D. Neumayer, V. Perebeinos, P. Avouris*, IBM T.J. Watson Research Center

Graphene is very promising for electronic devices, particularly for analogue high frequency devices due to its high intrinsic mobility. One challenge in graphene devices is the formation of gate dielectrics on graphene due to its hydrophobic nature. We found a new CVD gate insulator that provides uniform coverage on graphene while preserving the channel mobility, thus making it a very promising gate dielectric for graphene devices. We also studied the channel resistance and the modification of the band structure as a function of the vertical electric field near the Dirac (neutrality) points in the fabricated top-gated graphene devices. We found that as the vertical field increases, in bi- and tri-layer graphenes, the band-gap increases, while in multi-layer graphene, the band-overlap increases. The values of band-gap/overlap in bi-, tri- and multi-layer graphenes were estimated using a simple model which takes into account the variations of the surface electrostatic potential at the Dirac/neutrality point.

9:40am **GR+EM+MS+TF+MI-WeM6 Characterization of Devices Fabricated from Electrostatically Transferred Graphene: Comparison with Epitaxial based Devices**, *S.W. Howell, L.B. Biedermann, T. Ohta, T.E. Beechem, W. Pan, A.J. Ross, D.C. Trotter*, Sandia National Laboratories

10:40am **GR+EM+MS+TF+MI-WeM9 Electron Transport in Dual-Gated Mono- and Bilayer Graphene Devices with High-k Dielectrics**, *E. Tutuc, S. Kim, B. Fallah, K. Lee, J. Nah, S.K. Banerjee*, The University of Texas at Austin, *L. Colombo*, Texas Instruments, Inc.  
**INVITED**

A key issue for graphene-based devices is the deposition of thin high-k dielectric layers combined with a minimum electron mobility degradation. Here we examine the carrier transport in mono and bilayer graphene field-effect transistors with top Al<sub>2</sub>O<sub>3</sub> and HfO<sub>2</sub> dielectrics. The high-k dielectric films are grown by atomic layer deposition (ALD), and using a 1nm-thick Al interfacial layer in order to create intentional nucleation centers for the ALD. We show that this deposition technique allows the realization of high mobility graphene devices with aggressively scaled top dielectrics. We investigate the carrier mobility dependence on dielectric thickness and temperature in mono-layer graphene field-effect transistors with high-k dielectrics. The electron transport in dual-gated graphene bilayers with Al<sub>2</sub>O<sub>3</sub> top dielectric reveals an interesting band-gap energy dependence on transverse electric field and perpendicular magnetic field in this system.

11:20am **GR+EM+MS+TF+MI-WeM11 In-situ X-ray Photoelectron Spectroscopy Studies of Ozone-based ALD Al<sub>2</sub>O<sub>3</sub> Dielectrics on Graphite and Graphene**, *A. Pirkle, S. McDonnell*, University of Texas at Dallas, *L. Colombo*, Texas Instruments, Inc., *R.M. Wallace*, University of Texas at Dallas

We present a study of ozone-based atomic layer deposition (ALD) of Al<sub>2</sub>O<sub>3</sub> films on graphene and bulk graphite. Uniform deposition of scalable device-quality high-k dielectrics on graphene is a substantial hurdle for the implementation of conventional FET devices as well as novel device

structures exploiting the unique transport properties of graphene. Trimethylaluminum (TMA) / O<sub>3</sub> processes are found to result in uniform Al<sub>2</sub>O<sub>3</sub> depositions on graphite and graphene surfaces (1), in contrast to common TMA / H<sub>2</sub>O-based processes which result in nonuniform nucleation at defects and step edges.

In order to further examine the nature of interactions between TMA / O<sub>3</sub> and graphene, we utilize *in-situ* x-ray photoelectron spectroscopy (XPS) coupled via a UHV transfer line to an ALD reactor. Morphology of deposited films is also examined *ex-situ* using atomic force microscopy (AFM). We examine the impact of several parameters on Al<sub>2</sub>O<sub>3</sub> deposition. Choice of deposition temperature is critical, as etching of graphene by O<sub>3</sub> is observed at elevated temperatures (2) but dielectric quality is degraded at low temperature (3). We also examine the impact of surface condition on Al<sub>2</sub>O<sub>3</sub> composition particularly with regard to partially reacted TMA precursor molecules; various surface treatments are employed to approximate realistic device processing conditions. Finally, the effect of variations in purge time between ALD precursor pulses is studied; a reduction in deposition with increased purge time indicates that weakly bonded precursor molecules (TMA and O<sub>3</sub>) are easily desorbed from the graphene surface.

This work is supported by the NRI SWAN and MIND centers.

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2: G. Lee, et. al., J. Phys. Chem. C 113 (32), 14225 (2009)

3: S. K. Kim, et. al., J. Electrochem. Soc. 153 (5), F69 (2006)

# Wednesday Afternoon, October 20, 2010

## Graphene Focus Topic

Room: Brazos - Session GR+MS-WeA

### Low Dimensional Carbon Device Manufacturing

Moderator: A.C. Diebold, The University at Albany-SUNY

2:00pm **GR+MS-WeA1 Material Properties of Epitaxial Graphene in RF Devices**, *D.K. Gaskill*, U.S. Naval Research Laboratory, *J.S. Moon*, HRL Laboratories, LLC, *G.G. Jernigan*, *J.C. Culbertson*, *J.L. Tedesco*, U.S. Naval Research Laboratory, *J. Robinson*, The Pennsylvania State University, *P.M. Campbell*, *N. Garces*, *V.D. Wheeler*, *J.K. Hite*, *R.L. Myers-Ward*, *C.R. Eddy, Jr.*, *A.L. Friedman*, U.S. Naval Research Laboratory, *M. Fanton*, The Pennsylvania State University **INVITED**

The advent of the world's first epitaxial graphene (EG) RF field effect transistors (FETs), grown on semi-insulating SiC wafers, has generated tremendous interest in the electronics community since devices can be fabricated using conventional photolithographic approaches [1]. Recently, RF FETs have shown an  $f_{max}$  of 14 GHz at 5 V<sub>ds</sub> for a 2 μm gate width and better results are expected as gate widths are scaled down. To push the performance metrics for wafer-scale EG FETs significantly higher, key materials issues must be addressed. Some of these issues are morphology and thickness control, enhanced mobility, uniformity of sheet carrier density and resistivity, and substrate defects. Here we describe NRL-HRL-Penn State approach in the DARPA CERA program for forming EG via Si sublimation from SiC wafers and the impact of material issues on RF device performance will be discussed.

Epitaxial graphene was synthesized using an Aixtron VP508 reactor on the Si- and C-faces of 4H- and 6H-SiC semi-insulating 0° oriented substrates from 1225 to 1700°C and for 10 to 300 min. Samples were 50.8 and 76.2 mm wafers and 16 x 16 mm<sup>2</sup> witnesses. Both *in-vacuo* (10<sup>-6</sup> to 10<sup>-4</sup> mbar) and Ar ambient (50-200 mbar) sublimation conditions were investigated. Growth conditions resulted in continuous EG on Si-face witnesses < 1 nm thick as measured by atomic force microscopy, x-ray photoelectron spectroscopy and Raman spectroscopy, whereas growth on C-face witnesses could be varied, depending upon growth conditions, from island formation to continuous sheets > 10 nm thick. Using the witness samples, 300 K mobilities over 2,100 and 27,000 cm<sup>2</sup>V<sup>-1</sup>s<sup>-1</sup> were found for 10x10 μm<sup>2</sup> sized Hall patterns for EG on the Si- and C-face of SiC, respectively.

The growth of EG on 50.8 mm Si-face wafers resulted in excellent relative resistivity uniformity of 2.8% and 300 K Hall mobilities up to 2,700 cm<sup>2</sup>V<sup>-1</sup>s<sup>-1</sup> were found. Raman spectroscopy mapping of the 2D peak on the wafers determined: (1) the majority of the film was monolayer, (2) two layers of EG could be found at step edges and (3) EG was continuous across the wafer. RF FETs exhibited state-of-the-art ambipolar behavior such as electron field-effect mobility of 6,000 cm<sup>2</sup>V<sup>-1</sup>s<sup>-1</sup> with I<sub>on</sub>/I<sub>off</sub> ratio of 19 and peak transconductance of 600 mS mm<sup>-1</sup> per 1 fF μm<sup>2</sup> gate oxide capacitance. The f<sub>T</sub>•L<sub>g</sub> performance metric of 10 GHz•μm was established. Additionally, we will discuss our recent work on EG growth on 76.2 mm wafers as well as the impact of morphological features and Ar ambient controlled graphenization on future RF devices.

Supported by DARPA CERA (N66001-08-C-2048) and ONR.

[1] J.S.Moon *et al.*, IEEE Electron Dev Lett **31**, 260 (2010)

2:40pm **GR+MS-WeA3 In situ Growth, Microscopy, and Spectroscopy of Graphene Films**, *J. Hannon*, IBM T.J. Watson Research Center **INVITED**

I will describe *in situ* low-energy electron microscopy (LEEM) studies of graphene synthesis on SiC and polycrystalline Ni substrates. Using spatially-resolved electron diffraction (LEED-IV), we have determined the atomic structure, layer thickness, and stacking sequence of individual graphene domains with sub-micron precision. Using spatially-resolved electron energy loss spectroscopy (EELS), we have correlated the local electronic and atomic structure. I will discuss how these measurements aid in interpreting transport measurements from these same samples. This work was performed in collaboration with Ruud Tromp.

4:00pm **GR+MS-WeA7 High-frequency Transistors from Wafer-scale Epitaxial Graphene**, *Y.-M. Lin*, IBM T.J. Watson Research Center **INVITED**

Graphene has generated enormous research interest because of its unique physical and electronic properties. In particular, a large part of the research interests and activities arise from the high intrinsic carrier mobility and saturation velocity in graphene that may lead to higher-frequency electronic devices/circuits than can be achieved by conventional semiconductor

materials. Here we present the top-gated graphene transistors fabricated on two-inch graphene wafer.

Graphene was epitaxially grown on the Si face of a high-purity semi-insulating SiC 4H(0001) wafer by thermal decomposition, yielding a film of 1-2 layers of graphene over the entire wafer. The as-grown graphene film possesses an electron (*n*-type) carrier density of ~ 3x10<sup>12</sup> cm<sup>-2</sup> and a Hall-effect mobility between 1000–1500 cm<sup>2</sup>/V.s. In order to preserve the intrinsic mobility of graphene in the top-gated device structure, an interfacial polymer layer was spin-coated on the graphene prior to the oxide deposition. The carrier mobility of top-gated Hall bar devices varied between 900-1520 cm<sup>2</sup>/V.s across the two-inch wafer, indicating that little degradation in graphene mobility.

The cutoff frequency *f*<sub>T</sub> is obtained from the high-frequency S-parameters measurements, which signifies the highest frequency at which a transistor can propagate an electrical signal. For a gate length of 550 nm, the measured *f*<sub>T</sub> ranges between 20 to 53 GHz. For a shorter gate length of 240 nm, *f*<sub>T</sub> as high as 100 GHz was measured. This 100 GHz cutoff frequency is the highest speed achieved to date for any type of graphene devices, including exfoliated and CVD -grown graphene. Further enhancement in the device performance is expected to be achieved by continued improvements in the electrical characteristics of epitaxial graphene and the gate length scaling.

\*In collaboration with K. Jenkins, D. Farmer, C. Dimitrakopoulos, H.-Y. Chiu, A. Valdes-Garcia, A. Grill, and P. Avouris.

4:40pm **GR+MS-WeA9 Graphene-on-SiC and Graphene-on-Si MOSFETs on 75 mm Wafers**, *J.S. Moon*, *D. Curtis*, *M. Hu*, *S. Bui*, *D. Wheeler*, *T. Marshall*, HRL Laboratories, LLC, *D.K. Gaskill*, *P.M. Campbell*, Naval Research Laboratory, *P. Asbeck*, University of California at San Diego, *G.G. Jernigan*, *J.L. Tedesco*, *R.L. Myers-Ward*, *C. Eddy Jr.*, Naval Research Laboratory, *X. Weng*, *J. Robinson*, *M. Fanton*, Penn State University **INVITED**

In this talk, we present recent progress in epitaxial graphene n-MOSFETs and p-MOSFETs on both SiC and Si substrates for graphene-on-SiC and graphene-on-Si technologies. Both graphene MOSFETs were fabricated in a self-aligned manner on 75 mm wafers and exhibited gate-controlled ambipolar characteristics. For the graphene MOSFETs on SiC substrates, the graphene was grown by Si-sublimation of Si-face 6H-SiC substrates in a commercial Aixtron VP508 epitaxial reactor. For the graphene MOSFETs on Si substrates, the graphene was synthesized by graphitizing a thin 3C-SiC layer grown on float-zone Si (111) substrates using a halogen process. Figure 1 shows sheet resistance maps of 3-inch graphene-on-SiC and graphene-on-Si wafers. Typical Hall mobility ranges from 500 to 2000 cm<sup>2</sup>/Vs depending on electron carrier density. Both graphene MOSFETs were fabricated with a gate oxide layer and metal gate stack. The gate length was 3 μm. The graphene-on-SiC MOSFETs showed excellent I-V saturation behavior as shown in Figure 2(a). Figure 2(b) shows measured ambipolar behaviors with n-type MOSFET at V<sub>gs</sub> = 0 V, while p-type behaviors are observed at V<sub>gs</sub> < -1.5 V. An I<sub>on</sub>/I<sub>off</sub> ratio of 33 was measured. Figure 2(c) shows measured peak transconductance of 600 mS/mm at V<sub>ds</sub> = 3 V. Figure 3 shows the extrinsic field-effect mobility of 6000 cm<sup>2</sup>/Vs for electron and of 3200 cm<sup>2</sup>/Vs for hole obtained at an effective electric field of ~0.27 MV/cm, approaching Dirac point. The measured graphene field-effect mobility is eight to 10 times higher than that of ITRS Si n-MOSFETs and ~80 times higher than that of ultra-thin-body SOI n-MOSFETs.

The graphene-on-Si MOSFETs are fabricated in a similar manner. Figure 4 shows measured transfer curves of graphene-on-Si MOSFETs, showing ambipolar behaviors with the Dirac point close to zero gate bias, unlike the graphene-on-SiC MOSFETs. The on-state current is measured at 50 to 125 mA/mm with I<sub>on</sub>/I<sub>off</sub> ratio of 3 to 2, respectively. This is the highest performance observed among graphene-on-Si technologies so far. RF performance of graphene FETs will be discussed. This work was supported by DARPA, monitored by Dr. M. Fritze, under SPAWAR contract number N66001-08-C-2048.

The views, opinions, and/or findings contained in this article/presentation are those of the author/presenter and should not be interpreted as representing the official views or policies, either expressed or implied, of the Defense Advanced Research Projects Agency or the Department of Defense.

[1] C. Berger *et al.*, Science, vol. 312, p. 1191, 2006; J.S. Moon *et al.*, IEEE EDL, vol 30, p650, 2009





# Thursday Afternoon, October 21, 2010

## Spectroscopic Ellipsometry Focus Topic

Room: Cochiti - Session EL+AS+EM+MS+TF-ThA

## Spectroscopic Ellipsometry

Moderator: A.M. Creatore, Eindhoven University of Technology, the Netherlands

2:00pm **EL+AS+EM+MS+TF-ThA1 Developments in Spectroscopic Ellipsometry for Characterization of Organic and Inorganic Surfaces, Interfaces and Complex Layered Materials**, *M. Schubert*, University of Nebraska - Lincoln **INVITED**

In this paper we will review new developments in Spectroscopic Ellipsometry for characterization of organic and inorganic surfaces, interfaces and complex layered materials. Ellipsometry has matured over the past two decades with instrumentation and methodology capable of addressing today's and tomorrow's challenges in materials characterization and metrology. Ellipsometry measures the general state of polarization of light reflected or transmitted from samples. Owing to its nondestructive and generally applicable concept of investigating light emerging from specimens under investigations, and owing to its extreme accuracy and precision, ellipsometry has paved the way for almost all our modern technologies, continues to enable next-generation devices in electronics and optoelectronics, and emerges into fields of chemical, biochemical and biological platform technologies. Originating from the identification that the information carried upon the polarization within a light beam emerging from surfaces is extremely sensitive to mono and submonolayer characteristics, ellipsometry started decades ago pioneering development of microprocessors and electronic devices, which still today are based on planar thin film technology. Without ellipsometry, today's computation and information technology would be still in its infancy. In this paper, emerging developments and applications for metrology of optical and electrical properties of semiconductors and nanostructures by Terahertz Magneto-optic generalized ellipsometry, also referred to as the Optical Hall effect, will be highlighted. Examples will include state-of-the-art nitride semiconductor device structures and epitaxial graphene, candidates for tomorrow's next-generation devices. Likewise, new approaches for characterizing precise structural, magnetic and optical properties of three-dimensional nanostructure hybrid materials will be discussed. Examples will describe how ellipsometry characterization enables understanding and tailoring of electromagnetic properties of materials created by human intelligence, rather than by nature. New horizons are being explored currently by combining ellipsometry with independent surface sensitive techniques, such as acoustic Quartz-Crystal microbalance techniques. Combinations allow for identification of new information not accessible otherwise. Examples include observation of in-situ formation of self-assembled monolayers, protein adsorption onto sensitized surfaces, and formation of micelle-assisted bilayer configurations. Prospects, challenges and future developments will be reviewed from today's perspective.

2:40pm **EL+AS+EM+MS+TF-ThA3 Characterizing the Adsorption – Desorption Behavior of Organic Molecules Within Thin Mesoporous Carbon Composite Films using Spectroscopic Ellipsometry**, *B.D. Vogt, L.Y. Song, M.Z. Dai*, Arizona State University

Porous carbon materials are commonly utilized as adsorbants (i.e. activated carbon) and as catalyst supports. Direct templated synthetic routes to form ordered mesoporous carbons have recently been developed. By utilizing these concepts, mesoporous carbon composite films containing metal oxides can be synthesized. As a wide range of metal oxides can be utilized, these materials could be utilized in chemical sensing applications or as catalysts in fuel cell membranes. For both of these applications, the porous material will be exposed to organic vapors (such as ethanol in fuel cells). The condensation and evaporation of organic vapors from these materials is therefore an important consideration for their ultimate utilization in these applications. In-situ spectroscopic ellipsometry measurements of the mesoporous films exposed to controlled vapor pressures of organic vapors such as toluene, hexane and ethanol are utilized to understand the adsorption-desorption behavior of these films. Activated desorption of all three compounds is observed for pure carbon films, but addition of a small fraction of metal oxide enables the organics to be desorbed for the porous framework. These adsorption-desorption isotherms can also be utilized to estimate the pore size distribution and porosity of these films.

3:00pm **EL+AS+EM+MS+TF-ThA4 Mueller-Matrix Ellipsometry Studies of Chirality in Chitin-Based Structures and Thin Films of  $Al_{1-x}In_xN$** , *K. Järrendahl, H. Arwin, R. Magnusson, P. Sandström, C.-L. Hsiao, J. Landin, S. Vallyukh, J. Birch*, Linköping University, Sweden

A limited number of natural structures are known to reflect light that has circular or near circular polarization. This is for instance, the case for some scarab beetles where it is suggested that the polarization is caused by chiral structures in the form of helicoids in the cuticles. In this study, Mueller-matrix spectroscopic ellipsometry is applied in the spectral range of 250 to 1000 nm to investigate optical response and structures of the cuticle of various scarab beetles of the Cetoniinae subfamily. We will present our measurements showing how the polarization changes with wavelength as well as incidence angle and specify the conditions for when the reflected light is circularly left- or right-polarized. In most cases the reflected light is left-polarized as described by negative values of the M41 Mueller matrix element. For *Cetonia aurata*, a green beetle with metallic appearance, this is clearly seen in a rather narrow spectral range (470-550 nm). For other beetles (*Potosia cuprea* and *Licola lugubris*) similar polarization behavior is observed but the polarization features occur in a broader spectral region. We will show that there are even beetles (e.g. *Plusiotis argentiola*) reflecting both left- (M41 < 0) and right-polarized light (M41 > 0) in different parts of the spectral region. The Mueller data, including observations of the degree of polarization, are used to obtain structural and optical parameters from model calculations.

Our attempts to fabricate artificial structures with similar polarization properties will also be presented.  $Al_{1-x}In_xN$  thin films were grown on sapphire substrates by magnetron sputtering of indium and aluminum in a nitride atmosphere. Utilization of different seed layers and a substrate rotation gave chiral structures constituted by layers with a compositional gradient. Mueller-matrix results from these structures will be compared with the results from the natural structures. The Mueller data is also in this case very rich on information. In the initial steps to model these samples a similar approach as for the natural structures has been employed. Similarities and differences of the natural and artificial polarization response will be discussed in detail.

3:40pm **EL+AS+EM+MS+TF-ThA6 Mueller Polarimetry as a Tool for the Evaluation of the Diffraction Grating Profile Asymmetry**, *T. Novikova, P. Bulkin*, LPICM, CNRS, Ecole Polytechnique, France, *V. Popov*, Moscow State University, Russia, *A. De Martino*, LPICM, CNRS, Ecole Polytechnique, France

Mueller polarimetry in conical diffraction has proved to be a powerful optical technique for the metrological characterization of diffraction gratings. It was already shown that the shape of grating profile can be successfully reconstructed via appropriate optical modeling using full Mueller matrix measurements [1]. We also demonstrated that this approach can be of particular interest in microelectronics technology for the detection of overlay errors, which frequently result from the alignment deficiencies in lithography [2]. In some cases the asymmetrical distortion of grating profile can be induced by the etch process, or even be intentional, like in blazed gratings fabrication. For these applications a technique that allows for fast non-contact evaluation of the profile asymmetry may be of great value.

We studied the Mueller matrix spectra of symmetrical [3] and asymmetrical photoresist diffraction gratings on chromium using MM16 spectroscopic polarimeter, commercially produced by Horiba Jobin-Yvon, in the most general geometry of conical diffraction. At this configuration the 0th order cross-polarization complex reflection coefficients are antisymmetrical ( $r_{sp}^0 = -r_{ps}^0$ ), provided that the grating is composed of only reciprocal materials and is invariant under the rotation by  $180^\circ$  about the normal incidence [4]. It leads to the following relations between the elements of  $2 \times 2$  off-diagonal blocks of Mueller matrix:  $M_{ij} = \pm M_{ji}$ .

The lack of rotational symmetry violates the electromagnetic reciprocity theorem for the 0th-order diffraction on the asymmetrical gratings and, consequently, breaks the symmetry of the off-diagonal blocks of Mueller matrix ( $|M_{ij}|$  is not equal to  $|M_{ji}|$ ). This property of Mueller matrix of asymmetrical gratings was experimentally observed and numerically modeled at any illumination condition with exception of planar and pure conical mounting. We showed that the non-reciprocity in diffraction gratings can be used for the unambiguous detection of the grating profile asymmetry. The optimal choice of measurement configuration, i.e. azimuthal and polar angles considerably increases the sensitivity of the above mentioned technique.

References:

[1] T. Novikova, A. De Martino, S. Ben Hatit, and B. Drévilion, Appl. Opt. **45**, 3688 (2006).

[2] T. Novikova, A. De Martino, R. Ossikovski and B. Drévilion, *Europ. Phys. J. Appl. Phys.* **31**, 63 (2005).

[3] T. Novikova, A. De Martino, P. Bulkin, Q. Nguyen, B. Drévilion, V. Popov, and A. Chumakov, *Opt. Express* **15**, 2033 (2007).

[4] L. Li, *Opt. Soc. Am. A* **17**, 881 (2000).

**4:00pm EL+AS+EM+MS+TF-ThA7 Monitoring Ultra-Thin Organic Film Growth, In-Situ, with Combined Quartz Crystal Microbalance and Spectroscopic Ellipsometry, K.B. Rodenhausen, B.A. Duensing, A.K. Pannier, M. Schubert, University of Nebraska-Lincoln, M. Solinsky, The Procter & Gamble Company, T.E. Tiwald, J. A. Woollam Co., Inc.**

We report a combinatorial approach to study ultra-thin organic films. This novel technique consists of *in-situ* spectroscopic ellipsometry and quartz crystal microbalance methods. In contrast to the quartz crystal microbalance, which is sensitive to the total mass attached to the surface, including the trapped solvent, spectroscopic ellipsometry only measures the amount of adsorbent on the surface. We also introduce a new "virtual separation approach" ( $2\pi nd/\lambda \ll 1$ ) of analysis for the ellipsometry measurements. By using these two techniques in tandem, we are able to determine the thickness and solvent fraction of viscoelastic thin films.

We investigate cetyltrimethylammonium bromide (CTAB) thin films deposited onto a gold-coated quartz crystal as a model system. CTAB grown from a 2.5 mM solution demonstrates several phases in porosity evolution, including a temporary hold in water fraction as the film is rinsed off the substrate with water; these effects may be related to the structure of a CTAB bilayer.

In addition, a variety of self-assembled monolayers (SAMs) of alkanethiols on gold-coated quartz crystals are used as model biomaterials to determine the water fraction of an adsorbed prion layer. The porosity information distinguishes the proteins' conformation, dictated by the defined surface chemistries of the SAMs.

**4:20pm EL+AS+EM+MS+TF-ThA8 Ellipsometric Studies of Electronically Coupled PbSe and PbS Quantum Dot Thin Films, S.G. Choi, National Renewable Energy Laboratory, O.E. Semonin, University of Colorado, J.M. Luther, M.C. Beard, A.G. Norman, National Renewable Energy Laboratory, Z. Lin, Colorado School of Mines, A. Franceschetti, National Renewable Energy Laboratory, M.T. Lusk, Colorado School of Mines, A.J. Nozik, National Renewable Energy Laboratory**

Discovery of multiple exciton generation from colloidal suspensions of semiconductor quantum dots (QDs) has generated growing interests in realization of high-efficiency QD-based solar cells. Among a number of semiconductor QDs explored up to date, lead chalcogenides such as PbSe and PbS have been of great interest as a result of their wide tuning range of bandgap energy, abundance of materials, and large exciton Bohr radius.

In this presentation, I discuss optical properties of electronically coupled PbSe and PbS QD thin films. A series of QD multilayer thin films were prepared by a layer-by-layer dip-coating method onto glass substrates. Diameter of the QDs varies from 3.2 to 7.2 nm and from 3.5 to 8.3 nm for PbSe and PbS, respectively. Room-temperature pseudo-optical functions of the samples were measured by a rotating compensator-type, variable-angle spectroscopic ellipsometer. Transmittance data were also acquired in a normal-incidence configuration.

First, I determined refractive index  $N = n + ik$  of the QD films using the B-spline basis functions within the multilayer model (ambient/surface roughness/QD film/substrate). We use the  $N$  obtained as the input parameters for modeling the internal quantum efficiency of the QD-based solar cell devices. Then, I extracted dielectric function  $\epsilon = \epsilon_1 + i\epsilon_2$  for the ensemble of electronically coupled QDs using the Maxwell-Garnett effective medium approximations. The  $\epsilon$  spectra show the first exciton peaks, and the  $E_1$  and  $E_2$  critical-point (CP) structures whose energies are higher than the corresponding bulk values probably due to the quantum confinement effects. This abstract is subject to government rights.

**4:40pm EL+AS+EM+MS+TF-ThA9 In-situ Temperature Measurements by Spectroscopic Ellipsometry: Application to a-Si based Thin Films, D. Daineka, LPICM, CNRS, Ecole Polytechnique, France, V. Suendo, Institut Teknologi Bandung, Indonesia, P. Roca i Cabarrocas, LPICM, CNRS, Ecole Polytechnique, France**

Accurate measurement of the substrate temperature is of crucial importance in many semiconductor technologies such as plasma enhanced chemical vapor deposition (PECVD). Traditional tools, both thermocouples and pyrometers, are not always reliable for in situ measurements in vacuum when the substrate can be out of thermal equilibrium. On the other hand, non-contacting optical methods allow to determine the surface temperature with great accuracy, provided the temperature dependence of optical constants for the studied material is known. Since recently, spectroscopic ellipsometers are widely available and often installed on the research

deposition systems, which provides an opportunity to use them for temperature monitoring. We have studied the optical functions of amorphous silicon based thin films with spectroscopic ellipsometry in the temperature range from 290 to 520 K. The experimental data were modeled using Tauc-Lorentz dispersion law for amorphous materials. We have found that the temperature coefficients of Tauc-Lorentz parameters, such as the optical gap, are rather close for a few different materials. That similarity suggests that these values can be used to determine the surface temperature for a broad range of amorphous silicon based materials with a good accuracy. Practical examples of using spectroscopic ellipsometry for temperature measurements in the low pressure PECVD environment are given.

**5:00pm EL+AS+EM+MS+TF-ThA10 Real Time Spectroscopic Ellipsometry Studies of Amorphous and Nanocrystalline Si<sub>1-x</sub>Ge<sub>x</sub>:H Thin Films for Microbolometer Applications, D.B. Saint John, H.-B. Shin, M.-Y. Lee, E.C. Dickey, T.N. Jackson, N.J. Podraza, Penn State University**

Hydrogenated amorphous and nanocrystalline silicon (a/nc-Si:H), germanium (a/nc-Ge:H), and their alloys have been used and continue to be assessed for use in uncooled infrared microbolometer applications. These materials may be deposited as uniform layers using equipment common to the manufacturing of displays and photovoltaics and are thus more amenable to manufacturing considerations than the ion beam deposited vanadium oxide films used in most commercial microbolometers. Real optimization of material in the a/nc-Si<sub>1-x</sub>Ge<sub>x</sub>:H system for use in these devices requires a better understanding of the relationship between the key electrical properties of interest including resistivity ( $\rho$ ), temperature coefficient of resistance (TCR), and the 1/f noise character as a function of the degree of order and composition of the films. Si<sub>1-x</sub>Ge<sub>x</sub>:H thin films were deposited using plasma enhanced chemical vapor deposition using SiH<sub>4</sub>, GeH<sub>4</sub>, and H<sub>2</sub> at variable H<sub>2</sub>- dilution. These films have been monitored using in situ real time spectroscopic ellipsometry (RTSE) over a spectral range from 0.75 to 5.15 eV during deposition to detect changes in the film thickness and optical properties in the form of the complex dielectric function spectra ( $\epsilon = \epsilon_1 + i\epsilon_2$ ) as a function of deposition time. From the RTSE measurements and analysis it is possible to determine the structure of the material as amorphous, nanocrystalline, or mixed-phase and track the evolution of nanocrystallinity as a depth profile into the film. Ex situ Fourier transform infrared spectroscopic ellipsometry measurements over a spectral range from 0.05 to 0.75 eV were also performed to augment the complex dielectric function spectra and study absorption features relating to bonding. For electrical measurements, contacts were deposited in an isolated transfer length pattern for measurement of resistivity and TCR, while resistors with different volumes were made for volume normalization of the 1/f noise measurements. The TCR was measured from 20°C to 55°C. This study explores correlations between the electrical and optical properties of a-Si<sub>1-x</sub>Ge<sub>x</sub>:H and nc-Si<sub>1-x</sub>Ge<sub>x</sub>:H as functions of film processing conditions, resultant composition, and order. a-Si<sub>1-x</sub>Ge<sub>x</sub>:H films were prepared as a function of germanium content and hydrogen dilution to identify the impact that germanium and improved order at higher hydrogen dilution conditions have on the electrical properties ( $\rho$ , TCR, 1/f noise). The impact on the electrical properties due to the incorporation of small fractions of nanocrystallites are explored using mixed-phase (a+nc)-Ge:H films with nanocrystallite profiles guided by depth profile studies.

**5:20pm EL+AS+EM+MS+TF-ThA11 Roll-to-Roll Fabrication of Thin Film Si:H Solar Cells: Real Time Monitoring and Post Deposition Mapping by Spectroscopic Ellipsometry, L.R. Dahal, Z. Huang, D. Attygalle, M.N. Sestak, C. Salupo, S.X. Marsillac, R.W. Collins, University of Toledo**

## Spectroscopic Ellipsometry Focus Topic

Room: Southwest Exhibit Hall - Session

EL+AS+EM+MS+TF-ThP

## Spectroscopic Ellipsometry Focus Topic Poster Session

**EL+AS+EM+MS+TF-ThP1 Temperature Dependence of the Dielectric Function of AlSb Measured by Spectroscopic Ellipsometry, J.J. Yoon, Y.W. Jung, J.S. Byun, S.Y. Hwang, Y.D. Kim,** Kyung Hee University, Republic of Korea, **S.H. Shin, S.Y. Kim, J.D. Song,** Korea Institute of Science and Technology, Republic of Korea

AlSb is a promising material for applications in heterostructure devices such as long-wavelength detectors, quantum-well lasers, and laser diodes. However, to understand and properly design these devices, information about its electronic properties and its dielectric function  $\epsilon = \epsilon_1 + i\epsilon_2$  is needed. While room-temperature  $\epsilon$  data for AlSb exist, very little information is available about its behavior at elevated temperatures. Here, we report pseudodielectric function data  $\langle\epsilon\rangle$  from 300 to 800 K and from 0.7 to 5.0 eV, determined by spectroscopic ellipsometry. The samples were 1.5  $\mu\text{m}$  thick layers grown on GaAs (001) substrates by molecular beam epitaxy (MBE). This thickness significantly exceeds the critical value for AlSb, so the layers are fully relaxed. The MBE station features an integrated spectroscopic ellipsometer and strain-free windows, thereby allowing  $\epsilon$  data to be obtained without exposing the samples to air. For AlSb this is critical, because the removal of its oxides is not feasible owing to its reactivity. As a result of these precautions and the method by which these  $\langle\epsilon\rangle$  data were obtained, we consider them to be the most accurate representation of  $\epsilon$  to date. We also analyzed these data for critical-point (CP) parameters by fitting numerically calculated second energy derivatives of to standard analytic CP lineshape expressions. A parametric model was used, which describes dielectric functions by a combination of energy-bounded polynomials and poles, and encodes information in terms of amplitudes, critical-point energies, and broadening parameters. The reconstructed spectra are in excellent agreement with the data. We use these parameters to obtain information about the individual oscillators, including phonon effects, and interpolate them to obtain an analytic representation of the dielectric response of AlSb as a function of temperature. We expect these results to be an important database supporting engineering design, device technologies, and in-situ monitoring and control of device fabrication.

**EL+AS+EM+MS+TF-ThP2 Optical Properties and Humidity Effects on Thin Films of Micro Fibrillated Cellulose Studied by Spectroscopic Ellipsometry, H. Arwin, E. Antunez de Mayolo,** Linköping University, Sweden, **M. Eita,** Royal Institute of Technology (KTH), Sweden, **H. Granberg,** Innventia Ab, Sweden, **L. Wågberg,** Royal Institute of Technology (KTH), Sweden

High quality micro fibrillated cellulose (MFC) prepared from wood fibers can be used to prepare thin films on solid substrates by a layer-by-layer deposition technique. In applications MFC layers can be used alone or as a constituent in functional coatings, where the MFC can make a significant contribution to mechanical properties of the coating. Examples of potential applications are sensor layers, decorative coatings or mirrors. Here the optical properties in terms of the refractive index of MFC are studied with *in situ* spectroscopic ellipsometry (SE) and the effects on thickness and index due to humidity are investigated.

Films of MFC and polyethyleneimine (PEI) in the thickness range 30 - 300 nm are deposited on silicon substrates. Such MFC/PEI samples are exposed to water vapor in nitrogen (0-90% RH) and the ellipsometric response are measured *in situ* in the spectral range 245 - 1700 nm at an angle of incidence of 70 degrees using a dual-rotating compensator ellipsometer. From the SE-data the MFC/PEI film thickness and layer index are modeled.

Due to water exposure, the thickness of an MFC/PEI film is found to increase up to 15% or more, whereas the refractive index decreases. The effects are fully reversible and the dynamics of these changes are monitored with SE and are discussed.

**EL+AS+EM+MS+TF-ThP3 Spectroscopic Ellipsometry and X-ray Photoelectron Spectroscopy of La<sub>2</sub>O<sub>3</sub> Thin Films Deposited by Reactive Magnetron Sputtering, V. Atuchin,** Institute of Semiconductor Physics, Russia, **A.V. Kalinkin,** Boreskov Institute of Catalysis, Russia, **V.A. Kochubey, V.N. Kruchinin,** Institute of Semiconductor Physics, Russia, **R.S. Vemuri, C.V. Ramana,** University of Texas at El Paso

Lanthanum trioxide (La<sub>2</sub>O<sub>3</sub>) is one among the most promising high- $k$  dielectric materials to replace SiO<sub>2</sub> and Si<sub>3</sub>N<sub>4</sub> in advanced metal-oxide-

semiconductor devices in gate stack. La<sub>2</sub>O<sub>3</sub> can be prepared by various techniques but the film properties are strongly dependent on the fabrication conditions. Reactive magnetron sputtering deposition is widely used for the preparation of high quality transition multivalent metal oxide films with reproducible parameters and controlled thickness. The technique is preferred since it usually offers a high deposition rate for oxide films and a possibility to control the chemical composition of the film by reactive atmosphere in vacuum chamber. The aim of the present study is to understand the surface structure and evaluate the optical parameters of La<sub>2</sub>O<sub>3</sub> films deposited on Si substrates by magnetron sputtering. La<sub>2</sub>O<sub>3</sub> thin films were deposited onto Si(100) substrates in an argon/oxygen atmosphere using a high purity La target (99.9%). Structural parameters of the films were estimated by reflective high energy electron diffraction (RHEED) method at electron energy of 50 keV. All the films show no diffraction pattern indicating their amorphous nature in the near surface layers. Chemical state examined by the X-ray photoelectron spectroscopy (XPS), SPECS device, monochromatic Al K $\alpha$  radiation (1486.6 eV) before and after Ar<sup>+</sup> (2.5 keV, 2 min) sputtering indicates the stoichiometric film formation. Chemical nature of the species was identified with using binding energy (BE) difference parameter  $\text{DLA} = \text{BE}(\text{La } 3d_{5/2}) - \text{BE}(\text{O } 1s)$  [1]. Optical parameters of the films were measured with spectroscopic ellipsometry (SE) using a Spectroscan ellipsometer in the spectral range of 250 nm <  $\lambda$  < 1100 nm at the incidence angle of 70°. The La<sub>2</sub>O<sub>3</sub> films with thickness 500-850 nm were transparent over the spectral range and dispersion relations of refractive index  $n$  were well derived using a model of (air)/(single homogeneous layer)/(Si substrate). The curves  $n(\lambda)$  were approximated by Cauchy polynomials. Good relation between experimental points and theoretical curves confirms applicability of this simple model for the films. The optical parameters of La<sub>2</sub>O<sub>3</sub> defined with SE are related to film bulk and are insensitive to top surface effects induced by hydration or carbonate formation. These parameters will be used as a basis for SE evaluation of nanometric lanthanum oxide films with thickness below 10 nm.

1.V.V. Atuchin, T.A. Gavrilova, J.-C. Grivel, V.G. Kesler, Electronic structure of layered ferroelectric high- $k$  titanate La<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub>, J. Phys. D: Appl. Phys. 42 (2009) 035305.

**EL+AS+EM+MS+TF-ThP4 Analysis of Anomalous Film Growth when Yttrium Oxide Thin Films are Exposed to 7.2eV Light, D. Mortensen, D.D. Allred,** Brigham Young University

We have recently found that exposure of reactively sputtered yttrium oxide thin films to 7.2 eV photons in air produces a dramatic (factor of 4) increase in the films' thickness. This result was completely unexpected, Y<sub>2</sub>O<sub>3</sub> is exceptional stable, and demanded further investigation. This is particularly important since yttria and neighboring metal oxides such as ZrO<sub>2</sub> have been considered, and HfO<sub>2</sub> is being used, as components in gate oxides for silicon devices. The excimer lamp used for the study was of the type used in cleaning the surfaces of silicon wafers in the semiconductor industry. It is vital to understand how metal oxides might swell during cleaning.

We have experimentally observed the following:

1. The film-thickness increase is linear with exposure time up to a point.
2. Over a factor of four increase in film thicknesses, as measured by spectroscopic ellipsometry, has been observed. E.g., film which was about 20 nm thick as deposited reached a thickness of about 100nm.
3. When the yttrium oxide sample is placed in a furnace the changes rendered to the yttrium oxide sample are reversed.
4. A film placed in a plasma cleaner does not show this increase.
5. These effects are noticeably absent under the same conditions for a silicon wafer.
6. The refractive index of the film decreased with exposure time, suggesting the film could becoming more porous, though effective media optical models were unsuccessful in modeling the optical properties.

In addition to ellipsometry the films were studied by XPS and STEM.

**EL+AS+EM+MS+TF-ThP5 In-Situ Spectroscopic Ellipsometry of Lithium Ion Intercalation in GLAD Three-Dimensional Nanostructured Thin Films, E. Montgomery, M. Schubert, E.B. Schubert, T. Hofmann, D. Schmidt, University of Nebraska - Lincoln, R.A. May, University of Texas at Austin**

Lithium intercalation in thin nanostructured and bulk films from metals and semiconductors has been studied using in-situ spectroscopic ellipsometry. Nanostructured thin films have a large surface area, and the stress caused by the intercalation of lithium is reduced in comparison to continuous films. The films are deposited using electron beam evaporation at a glancing angle or Glancing Angle Deposition (GLAD).

The charge capacity of the films can be observed as a change of index of refraction using ellipsometry. We will study intercalation as a function of structure geometry in chiral and achiral 3D GLAD thin films deposited from different metals onto silicon and other dielectric substrates. We report the anisotropic dielectric functions of the 3D nanostructures and their changes as a function of intercalation time and period.

**EL+AS+EM+MS+TF-ThP6 Multi Phase Model Generation of Reflection Anisotropy Spectra of Copper Phthalocyanine Films on Vicinal Silicon Substrates, F. Seidel, L. Ding, O.D. Gordan, D.R.T. Zahn, Chemnitz University of Technology, Germany**

In this work the in-plane anisotropy of copper phthalocyanine (CuPc) thin films grown on vicinal silicon substrates is explained by simulating Reflection Anisotropy Spectroscopy (RAS). In RAS the complex difference in reflection along two perpendicular directions is measured at an incidence angle close to  $0^\circ$ . While RAS has strong similarities with another polarisation related measurement technique, Spectroscopic Ellipsometry (SE), simulations of RA spectra using a similar mathematical formalism like in SE is not common.

One difference is that in SE the measurements are usually performed at an incidence angle close to the Brewster angle, where the difference between s and p reflected polarisation is maximal. However due to the similarities between the two techniques the evaluation of RA spectra can be performed in a similar way like for SE after some simple mathematical transformations. This is particularly useful when thickness induced interference can lead to incorrect interpretations of RA spectra. Therefore in this work we show that the RA spectra evaluation of CuPc layer with increasing thickness is mainly given by the optical interference effect and not by a change in the optical anisotropy of the film.

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